

# PATENT SPECIFICATION

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## (54) PRODUCTION OF CUBIC BORON NITRIDE

(71) We, DE BEERS INDUSTRIAL DIAMOND DIVISION LIMITED, a Company registered according to the laws of the Republic of South Africa, of 45 Main Street, Johannesburg, Transvaal, Republic of South Africa, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to cubic boron nitride.

Cubic boron nitride is a hard substance, second only to diamond in hardness. The substance forms the subject of U.S. Patent Specification No. 2,947,617. Cubic boron nitride (CBN) is produced by subjecting hexagonal boron nitride in the presence of a suitable solvent/catalyst to conditions of elevated temperature and pressure at which cubic boron nitride is crystallographically stable. Cubic boron nitride is produced in the presence of a substance which acts as a solvent or catalyst or both. Such substances will hereinafter and in the claims be referred to simply as catalysts. Examples of suitable catalysts are given in the above mentioned U.S. Specification and include alkali metals, alkaline earth metals, lead, antimony, tin and nitrides of these metals. Other catalysts have been developed and aluminium/iron alloys may be given by way of example.

The production of cubic boron nitride requires the use of very high temperatures and pressures. Such can be generated in an apparatus known as the "belt" apparatus which forms the subject of U.S. Patent Specification No. 2,941,248. This apparatus consists essentially of an annular belt or die member having a tapering aperture therethrough and a pair of concentric frustoconical punches which are capable of moving into the aperture to define a reaction chamber or zone therein. A

suitable gasket material such as pyrophyllite is employed between the punches and die members for sealing purposes and over the inner surface of the die facing the reaction zone to insulate thermally this portion of the die. The temperature of the reaction chamber may be raised by connecting the punch members to a source of electrical power thereby creating a resistance heating circuit through the punches and the contents of the reaction chamber.

Cubic boron nitride is a good abrasive for the grinding of steels such as high speed steels. For abrading operations, it generally forms part of a resin bond grinding wheel.

According to one aspect of the present invention, there is provided an elongate cubic boron nitride particle having a long axis and a short transverse axis, the ratio of the long axis to the short axis being at least 3:1 and the long axis being in the <111> crystallographic direction.

According to another aspect of the present invention, a method of making elongate cubic boron nitride particles as defined in the last preceding paragraph, including the steps of providing a reaction zone placing in the reaction zone a sleeve of hexagonal boron nitride around a core of catalyst for boron nitride formation, the weight ratio of hexagonal boron nitride to catalyst being in the range 10:1 to 3:1, the catalyst being an alkali metal or alkaline earth metal or nitride thereof and subjecting the contents of the reaction zone to conditions of temperature and pressure suitable for cubic boron nitride formation.

Of course, some particles which are not of elongate shape are produced simultaneously with the elongate particles.

The conditions of temperature and pressure may be any known in the art for cubic boron nitride production. Examples of suitable temperatures and pressures may be found in the above-mentioned U.S.

Patent Specification No. 2,947,617. In general, however, the temperatures used will be in the range 1500°C to 2000°C and the pressures used will be in the range 50 kilobars to 100 kilobars. Preferred catalysts are the alkali and alkaline earth metal nitrides, particularly calcium nitride and lithium nitride.

The hexagonal boron nitride (HBN) is provided in the form of a sleeve around a core of the catalyst. It has been found that radial areas of weakness are produced in the HBN sleeve when it is subjected to the required temperature and pressure conditions and that the catalyst penetrates these areas of weakness to produce the elongate particles. The HBN sleeve having the catalyst core may be placed in a pyrophyllite sleeve and then in the reaction zone of a high temperature pressure apparatus in the conventional manner.

The sleeve of HBN may be a continuous sleeve or may consist of a plurality of segments which together define the sleeve. The core and sleeve are preferably dimensions for a snug fit one inside the other. The core may be in the form of loose powder, but is preferably compacted into a coherent body. Any suitable compacting method known in the art may be used. A disc of HBN may be provided at each of opposite ends of the core.

The weight ratio of hexagonal boron nitride to catalyst is preferably in the range 6:1 to 5:1.

Examples of the elongate cubic boron nitride particles of the invention are shown in the photographs attached as Figures 1 and 2. The magnification of the photograph as Figure 1 is 269 and of the photograph as Figure 2 is 273. It will be noted that the long axes of these particles are in the <111> crystallographic direction and the particles have an irregular surface.

In determining the ratio of long axis to short axis one takes the long and short axes of greatest dimension for this determination.

The particles may vary in size but will generally be in the range 60/200 U.S. mesh. The particles are preferably in the range 80/170 U.S. mesh.

The novel particles of the invention are generally friable rendering them particularly suitable for grinding operations where their friability allows for fresh cutting surfaces to be continually produced during the grinding operation. The particles may be incorporated into the abrading portion of abrasive tools such as resin bond and metal bond abrasive tools. It is preferred that the particles are so orientated in the abrading portion of the tool that their long axes are substantially normal to the working face.

Because of their friability, the particles are preferably used in resin bond grinding wheels. The irregular surface of the particles assists in keying the particles to the resin matrix. To improve this keying further, it is preferred that the particles are metal, preferably nickel, coated. For maximum effectiveness the particles, as mentioned above, are preferably so orientated in the grinding operation of the wheel that their long axes are substantially normal to the working face thereof.

Resin bond grinding wheels are well known in the art, as are their methods of manufacture. Briefly, resin bond grinding wheels are made by providing a suitable mould around a hub portion, generally of a material such as Bakelite (Registered Trade Mark), introducing a mixture of powdered resin starter ingredients, cubic boron nitride and filler into the mould, and applying pressure and heat to the mixture in the mould to cause the resin to cure and set. The resin may be a phenolformaldehyde or polyimide resin or any other resin known in the art of grinding wheels.

The amount of cubic boron nitride in the operating grinding portion of the wheel will vary according to the type of wheel. The cubic boron nitride content of the grinding portion will generally constitute about 10 to 25 volume percent.

The particles may be orientated by means of an impressed field of force using known techniques. The impressed field of force may be electrostatic. Alternatively, the particles may be coated with a magnetic material such as a ferromagnetic metal and the particles orientated by means of an externally impressed magnetic field.

In an example of the invention a core of compacted lithium nitride was placed inside a sleeve of hexagonal boron nitride. The dimensions of the core and sleeve were such that there was a snug fit. Hexagonal boron nitride discs were then placed on the top and bottom of the core. Figure 3 of the attached drawings illustrates schematically the arrangement. Referring to this figure, the core is shown at 10 and the sleeve and discs at 12 and 14, respectively. The weight ratio of the hexagonal boron nitride to lithium nitride was about 6:1.

The lithium nitride/HBN composite was then placed in a sleeve of pyrophyllite and the whole placed in the reaction zone of a high temperature/pressure apparatus of the type described in U.S. Specification No. 2,941,248.

The temperature and pressure of the reaction zone were raised to 1500°C and 55 kilobars by first raising the pressure, and then the temperature, to the desired values. These conditions were maintained for

about ten minutes and then released by first allowing the temperature, and then the pressure, to drop to ambient conditions. The cubic boron nitride content of the reaction capsule was recovered using conventional techniques. The cubic boron nitride produced contained a large percentage of elongate or needle-shaped particles of the type illustrated by Figures 1 and 2.

The elongate particles produced were mostly in the range 60/170 U.S. mesh.

WHAT WE CLAIM IS:—

1. An elongate cubic boron nitride particle having a long axis and a short transverse axis, the ratio of the long axis to the short axis being at least 3:1 and the long axis being in the  $\langle 111 \rangle$  crystallographic direction.

2. An elongate cubic boron nitride particle according to Claim 1, having an irregular surface.

3. An elongate cubic boron nitride particle according to Claim 1 or Claim 2, which is friable.

4. An elongate cubic boron nitride particle according to any one of Claims 1 to 3, which is in the range 60/200 U.S. mesh.

5. An elongate cubic boron nitride particle according to Claim 4, which is in the range 80/170 U.S. mesh.

6. An elongate cubic boron nitride particle according to any one of Claims 1 to 5 which is metal coated.

7. An elongate cubic boron nitride particle according to Claim 6, wherein the metal is nickel.

8. An elongate cubic boron nitride particle substantially as herein described

with reference to Figures 1 and 2 of the accompanying drawings.

9. A method of making elongate cubic boron nitride particles according to Claim 1, including the steps of providing a reaction zone placing in the reaction zone a sleeve of hexagonal boron nitride around a core of catalyst for boron nitride formation, the weight ratio of hexagonal boron nitride to catalyst being in the range 10:1 to 3:1, the catalyst being an alkali metal or alkaline earth metal or nitride thereof and subjecting the contents of the reaction to conditions of temperature and pressure suitable for cubic boron nitride formation.

10. A method according to Claim 9, wherein the weight ratio of the hexagonal boron nitride to catalyst is in the range 6:1 to 5:1.

11. A method according to Claim 9 or Claim 10, wherein the catalyst is selected from calcium nitride and lithium nitride.

12. A method according to any one of Claims 9 to 11, wherein the temperature is in the range 1500°C to 2000°C and the pressure is in the range 50 kilobars to 100 kilobars.

13. A method of making elongate cubic boron nitride particles substantially as herein described with reference to Figure 3 of the accompanying drawings.

14. Elongate cubic boron nitride particles made by a method according to any one of Claims 9 to 13.

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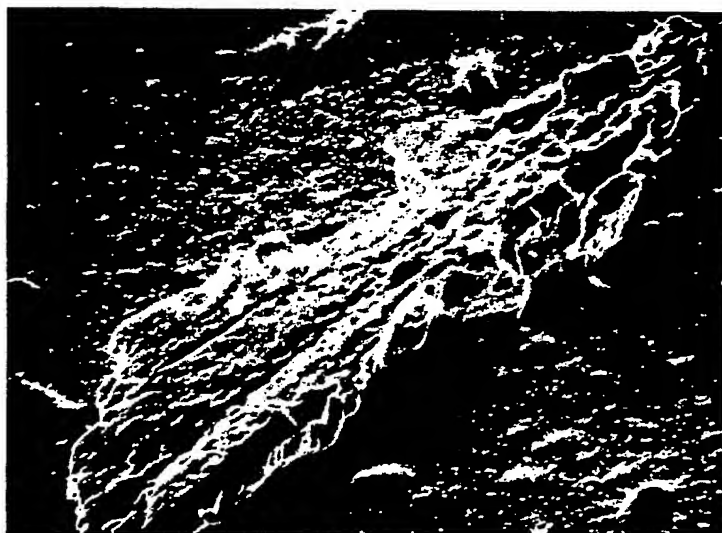
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COMPLETE SPECIFICATION

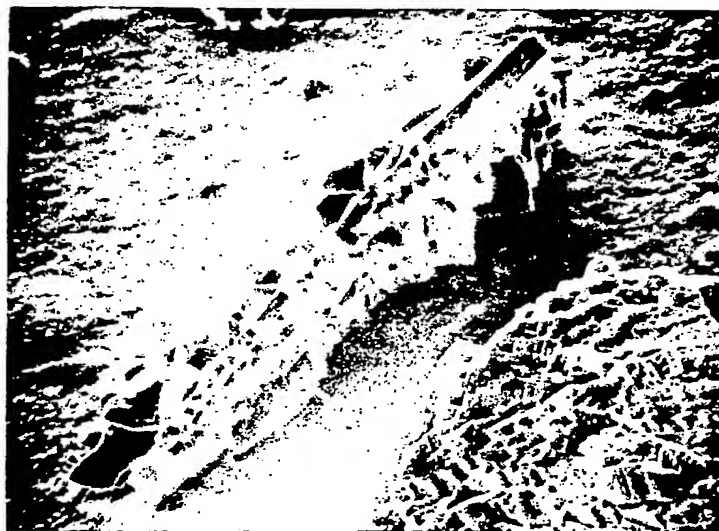
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SHEET 1



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